



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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In re application of:

Tetsuo KODAMA et al.:

Serial No. 09/502,834:

Group Art Unit: 1711

Filed on February 11, 2000:

Examiner: Duc Truong

For: POLYBENZAZOLE ARTICLE AND PRODUCTION METHOD THEREOF

DECLARATION UNDER 37 CFR 1.132

Honorable Commissioner of
Patents and Trademarks,
Washington, D.C. 20231

Sir:

I, Tetsuo KODAMA, whose full post office address is
c/o Toyo Boseki Kabushiki Kaisha, Research Center, 1-1,
Katata 2-chome, Ohtsu-shi, Shiga 520-0292 Japan, sincerely
declare:

That my education and employment history is as follows:

That I was graduated from Department of Marine Science,
Faculty of Marine Science and Technology, Tokai University
in March 1981,

That I completed the master course study of
Environmental Science at Hiroshima University, graduate
school (Faculty of Integrated Arts and Science) in March
1983,

In April 1983, I was employed by Toyo Boseki Kabushiki
Kaisha, and I have been engaged in the research and
development of base films of magnetic tapes, PPS, RO module,
PBO and so on at Toyo Boseki Analytical Research Center;

That I am one of the inventors of the above-identified
U.S. Patent Application No. 09/502,834 and familiar with the
subject matter of this invention;

That I have reviewed the Office Action dated May 7,
2003 issued in the above-identified application and have
directly conducted the following experiments to show that
the polybenzazole article of the present invention
comprising a light-resisting agent defined in claim 1 is
markedly superior in light resistance as compared to a
polybenzazole article comprising, as a light-resisting
agent, naphthol described in So et al. (USP No. 5,552,221);

That the following Experiments demonstrate such fact,
the results of which follow hereunder;

Experiments

(1) Object

To prepare the polybenzazole (PBO) article of the present invention comprising a light-resisting agent defined in claim 1 and a polybenzazole article comprising, as a light-resisting agent, naphthol described in So et al. (USP No. 5,552,221) and evaluate the light resistance thereof.

(2) Preparation method of samples for evaluation

(Preparation of PBO sample)

A spinning dope containing polyparaphenylene benzobisoxazole (14.0% by weight) obtained by the method described in USP 4,533,693 and having an intrinsic viscosity as measured with a methanesulfonic acid solution at 30°C, of 24.4 dL/g, and polyphosphoric acid containing phosphorus pentaoxide in 83.17% by weight was spun. The dope was passed through a metal net filter, and kneaded and defoamed in a twin screw kneader. The pressure was elevated, the dope temperature was maintained at 170°C, and the dope was spun at 170°C from a spinneret. The delivered yarns were cooled with cooling air at 60°C and wound around a godet roller to afford a spinning speed. The yarns were led into an extraction (coagulation) bath of a 20% by weight aqueous phosphoric acid solution maintained at a temperature of $20 \pm 2^\circ\text{C}$. The yarns were successively washed with ion exchange water in a second extraction bath, and immersed in a 0.1 mol/L sodium hydroxide solution for neutralization. The resulting undried PBO yarns were preserved in water and used as a sample.

(Preparation of treating solution for light-resisting agent)

Each light-resisting agent shown in the following Table 1 was dissolved in ethanol such that the concentration was 1 g/100 ml and the liquid temperature was about 40°C to give a treating solution. However, since Acid Fuchin, sodium salt has low solubility, the concentration was less than 1 g/100 ml.

(Treatment with light-resisting agent)

The undried PBO yarn (249d, 166f) obtained above was placed in each treating solution while preventing entanglement, and stood at about 40°C for 4 hr. Redundant treating solution was removed by pressing waste (JK wiper paper) and the yarn was dried to give a sample for evaluation.

(3) Evaluation of light resistance

The samples for evaluation (PBO fiber) obtained above were folded three times (for forming a kink band and accelerating the light resistance evaluation) and subjected to xenon light exposure at 83°C for 24 hr. The tensile strength of mainly the part folded 3 times was measured and strength retention was calculated with the tensile strength (DT 40.8 (g/d)) of dry PBO yarn samples free of treatment with a light-resisting agent as 100%.

(4) Evaluation results

The obtained strength retention is shown in the following Table 1.

Table 1

Sample	Light-resisting agent	Strength retention (%) after xenon light exposure for 24 hr
1	Blank (not treated)	24
2	Rhodamine B	26
3	Acid Fuchin, sodium salt	28
4	1-Naphthol	23
5	2-Naphthol	27
6	o-Aminophenol/p-Phenylenediamine=1/1	34
7	m-Phenylenediamine/p-Phenylenediamine=1/1	55

The light-resisting agent (Samples 6 and 7) of the present invention is oxidized in an aqueous solution to form a condensate (colors the aqueous solution black), reacts with or adsorbs to PBO yarn to dye the PBO yarn. By mixing two kinds of light-resisting agents, the

oxidation/condensation proceeds further to afford a striking light resistance effect (higher strength retention (%)).

(5) Conclusion

The polybenzazole articles (Samples 6 and 7) of the present invention comprising a light-resisting agent defined in claim 1 showed significantly higher strength retention (%) (dramatically superior in light resistance) after xenon light exposure for 24 hr, as compared to polybenzazole articles (Samples 2 - 5) comprising, as a light-resisting agent, a dye, particularly naphthol (Samples 4 and 5) described in So et al. (USP No. 5,552,221).

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at Shiga, Japan on this 29th day of October, 2003

Tetsuo Kodama

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Tetsuo KODAMA